

(12) **UK Patent Application** (19) **GB** (11) **2 048 960 A**

- (21) Application No 8013737  
(22) Date of filing 25 Apr 1980  
(30) Priority data  
(31) 2917348  
(32) 28 Apr 1979  
(33) Fed. Rep. of Germany (DE)  
(43) Application published  
17 Dec 1980

(51) INT CL<sup>3</sup>  
C23C 11/08

(52) Domestic classification  
C7F 1A 1B2 1B5 2M 2Z2  
4D 4K 4X

(56) Documents cited

GB 1544436  
GB 1509752  
GB 1494157  
GB 1488696  
GB 1425633  
GB 1394108  
GB 1389140  
GB 1386900  
GB 1297896

(58) Field of search  
C7F

(71) Applicant  
Fried Krupp Gesellschaft  
Mit Beschränkter Haftung,  
Altendorfer Strasse 103,  
D-4300 Essen 1, Federal  
Republic of Germany

(72) Inventors  
Klaus Dreyer,  
Hans Grewe,  
Johannes Kolaska,  
Norbert Reiter

(74) Agents  
Brewer & Son

**(54) Coating Articles With Layers of Hard Materials**

(57) A composite body, for use as a part resistant to wear or as an insert in a cutting tool, comprises a substrate carrying one or more surface layers of

hard material devoid of binder. The thickness of each such surface layer is 1 to 50  $\mu\text{m}$  and one of the layers is composed of a multiplicity of individual layers each having a thickness of 0.02 to 0.1  $\mu\text{m}$ . Alternate individual layers consist of hard material of different composition.

GB2 048 960A

# SPECIFICATION Composite Body

The invention relates to a composite body which consists of a substrate and one or more surface layers of hard material of different composition and devoid of binder metal. The substrate may consist, for example, of steel, of ceramic material or of hard metal consisting of at least one appropriate metal carbide as the hard material and at least one of the binder metals cobalt, iron or nickel. All layers of hard material constitute the hard coating of the substrate and each layer of hard material consists of one or more hard materials. The hard materials are carbides, nitrides, borides, silicides or oxides which have a high resistance to wear and high chemical stability. The known layers of hard material normally have a thickness of 1 to 50  $\mu\text{m}$ .

In published Austrian Patent Application A 6317/72 is described a composite body of hard metal intended for use as a wear resistant element which has, at least on the portions subjected to wear, a coating of hard materials which consists of several layers of different composition and consisting of compounds of elements of Groups III to VI of the Periodic System with carbon and/or nitrogen and/or boron and/or silicon, the individual layers having a thickness of 0.2 to 30  $\mu\text{m}$  and merging into one another over a thickness of 0.1 to 10  $\mu\text{m}$ . In addition, DE-AS 2,253,745 describes a composite body, which consists of a core and two superposed layers of hard material devoid of binder metal, the outer layer consisting of one or more extremely wear resistant deposits of aluminium oxide and/or zirconium oxide and having a thickness of 0.2 to 20  $\mu\text{m}$  while the inner layer adjoining the core consists of one or more carbides and/or nitrides of the elements titanium, zirconium, hafnium, vanadium, niobium, tantalum, chromium, molybdenum, tungsten, silicon and/or boron and has a thickness of 1 to 10  $\mu\text{m}$ . Also DEOS 2,525,185 describes wear-resistant parts consisting of a hard metal substrate and two superposed layers, the outer and extremely wear-resistant layer consisting of aluminium oxide and/or zirconium oxide and the inner layer consisting of one or more borides, in particular diborides of the elements titanium, zirconium, hafnium, vanadium, niobium, tantalum, chromium, molybdenum and tungsten, the inner layer and the outer layer each having a thickness of 0.5  $\mu\text{m}$  to 20  $\mu\text{m}$ . Finally, GB-PS 1,509,752 describes steel or hard metal tools coated with several layers of hard materials of different composition, the thickness of the coating being 1 to 50  $\mu\text{m}$  and the thickness of the individual layers being 0.5 to 20  $\mu\text{m}$ . The known composite bodies have the disadvantage that the coatings of hard material have inadequate adhesive strength when subjected to wear. In particular the oxide layers have a tendency to crack, and the cracks lead to a separation of the coatings.

The object of the invention is to provide a composite body in which the layers of hard material devoid of binder metal have besides a high wear resistance an improved adhesion to the substrate and to the adjoining layers, because the high wear resistance of the layers of hard material can only be fully utilized when they are bound sufficiently firmly to the substrate and to one another. In addition, the hard material coating should have a high fracture toughness and thus be able to resist higher stresses requiring toughness.

The problem underlying the invention is solved in that one of the layers of hard material in the composite body has a thickness of 1 to 50  $\mu\text{m}$  and is composed of a multiplicity of thin individual layers each having a thickness of 0.02 to 0.1  $\mu\text{m}$ , the hard material in each of these individual layers differing in composition from that in both of the adjoining individual layers. Surprisingly, it has been found that a composite body according to the invention has both a high wear resistance and a substantially greater adhesive strength of the entire coating of hard material, notwithstanding the fact that the coating can be made both of relatively thick layers and of a multiplicity of thin individual layers. In particular, it was not to be expected that when the composite body is subjected to wear, energy-consuming phenomena would occur in the layer consisting of a multiplicity of thin individual layers, which strongly inhibit formation of cracks and premature separation of the coating of hard material. The composite body according to the invention has, in addition, a high fracture toughness and, consequently, can resist higher stresses requiring toughness.

It has been found that the composite body according to the invention can take the following forms:—

- (a) Substrate—layer of hard material composed of many thin individual layers.
- (b) Substrate—one or more layers of hard material—layer of hard material composed of many thin individual layers.
- (c) Substrate—one or more layers of hard material—layer of hard material composed of many thin individual layers—one or more layers of hard material.
- (d) Substrate—layer of hard material composed of many thin individual layers—one or more layers of hard material.

The layers of hard material have a thickness of 1 to 50  $\mu\text{m}$ . As a further feature of the invention it is proposed that the layer of hard material composed of a multiplicity of individual layers has a thickness of 3 to 15  $\mu\text{m}$ , as this thickness provides optimum properties for the composite body. The composite body according to the invention has been found to be

particularly advantageous when the many thin individual layers consist alternatively of titanium carbide, nitride or carbonitride and of aluminium oxide or zirconium oxide, or, as another possibility, alternately of titanium carbide and zirconium carbonitride. It is also specially advantageous for the substrate of the composite body to be metallic, a substrate of steel or hard metal being especially suitable. According to a further feature of the invention a substrate of hard metal may carry a hard material layer of titanium carbide on which is a hard material layer consisting of a multiplicity of thin individual layers which are alternatively of titanium nitride and of aluminium oxide. Alternatively the hard metal substrate may carry a hard material layer, consisting of a multiplicity of thin individual layers consisting alternatively of titanium carbide, nitride or carbonitride and of aluminium or zirconium oxide, on which is a hard material layer of aluminium oxide. The outer layer of aluminium oxide may have a nitrogen content of 0.5 to 4 atom %. Owing to its high wear resistance, the composite body according to the invention may be used for elements subjected to wear or for machining metallic and non-metallic materials.

Various known coating procedures, in particular the CVD procedure, can be used to produce the composite body. The multiple thin individual layers can be identified by electron microscopy.

The invention will now be explained in more detail, by reference to an example. A substrate of hard metal, in the form of an indexable insert, was provided in a furnace at a temperature of 1000°C, at a pressure of 50 mbar and over a period of 100 minutes with a hard coating of titanium carbide having a thickness of 3 µm, the titanium carbide being produced by a vapour phase reaction from titanium tetrachloride, methane and hydrogen. Then, in the same furnace at a temperature of 1000°C, at a pressure of 50 mbar and over a period of 250 minutes there was deposited on the layer of titanium carbide a coating of hard material which had a thickness of 3 µm and consisted in alternation of 19 layers of titanium nitride and 19 layers of aluminium oxide.

The 38 individual layers were formed by vapour phase reactions, the individual layers of titanium nitride being formed using a gas composed, in proportions by volume, of 22.2% nitrogen, 1.2% titanium tetrachloride and 76.6% hydrogen and the layers of aluminium oxide being formed using a gas composed, in proportions by volume, of 4.0% carbon dioxide, 16.6% carbon monoxide, 2.3% aluminium chloride and 77.1% hydrogen. The resulting composite body was used as an indexable insert for machining metallic materials and found to have in comparison with known coated inserts a substantially higher wear resistance and higher toughness as shown by the following experimental results.

#### 1. Smooth cutting:

Material machined: Steel C 60

Form of the indexable insert in accordance with ISO 1832: SNUN 120408

Cutting speed: v=200 m/min

Depth of cutting x feed: ax=1.5x0.28 mm<sup>2</sup>/rev

Machining time: T=10 min.

		Crater depth KT µm	Flank wear VB mm	
40	Tool insert of hard metal carrying a layer of TiC of thickness 6 µm	46	0.20	40
45	Tool insert of hard metal carrying an inner layer of TiC of thickness 5 µm and an outer layer of Al <sub>2</sub> O <sub>3</sub> of thickness 1 µm	30	0.18	45
	Tool insert according to the invention of hard metal carrying an inner layer of TiC of thickness 3 µm and an outer layer of thickness 3 µm and consisting of 19 layers of TiN and 19 layers of Al <sub>2</sub> O <sub>3</sub>	10	0.13	

#### 2. Interrupted cutting:

Four rods of C 45 KN steel, having a diameter of 40 mm and a length of 60 mm, were clamped parallel to one another in holes disposed in a circle of diameter 190 mm in a supporting device and were surfaced in an outward direction.

Cutting speed: 250 m/min  
Depth of cut: a=2 mm  
Feed: s=0.22 mm/rev.

		<i>Number of cuts</i>	
	Tool insert of hard metal carrying a layer of TiC of thickness 6 $\mu\text{m}$	19200	
5	Tool insert of hard metal carrying an inner layer of TiC of thickness 5 $\mu\text{m}$ and an outer layer of $\text{Al}_2\text{O}_3$ of thickness 1 $\mu\text{m}$	17920	5
10	Tool insert according to the invention of hard metal carrying an inner layer of TiC of thickness 3 $\mu\text{m}$ and an outer layer of thickness 3 $\mu\text{m}$ and consisting of 19 layers of TiN and 19 layers of $\text{Al}_2\text{O}_3$	32000	10

#### Claims

1. A composite body which consists of a substrate and one or more layers of hard material of different composition and devoid of binder material and each layer having a thickness of 1 to 50  $\mu\text{m}$ , characterized in that one of the layers of hard material is composed of a multiplicity of thin individual layers each having a thickness of 0.02 to 0.1  $\mu\text{m}$ , the hard material in each of these individual layers differing in composition from that in both of the adjoining individual layers. 15
2. A composite body according to claim 1, characterized in that the layer of hard material composed of a multiplicity of individual layers has a thickness of 3 to 15  $\mu\text{m}$ .
3. A composite body according to claims 1 and 2, characterized in that the individual layers consist alternately of titanium carbide, nitride or carbonitride and of aluminium oxide or zirconium oxide. 20
4. A composite body according to claims 1 and 2, characterized in that the individual layers consist alternately of titanium carbide and of zirconium carbonitride.
5. A composite body according to claims 1 to 4, characterized in that it has a metallic substrate.
6. A composite body according to claims 1, 2, 3 and 5, characterized in that the substrate is of hard metal carrying a hard material layer of titanium carbide on which is a hard material layer consisting of a multiplicity of thin individual layers which are alternately of titanium nitride and of aluminium oxide. 25
7. A composite body according to claims 1, 2, 3 and 5, characterized in that the substrate is of hard metal carrying a hard material layer, consisting of a multiplicity of thin individual layers consisting alternately of titanium carbide, nitride or carbonitride and of aluminium or zirconium oxide, on which is a hard material layer of aluminium oxide. 30
8. A composite body according to claim 7, characterized in that the outermost hard material layer consists of aluminium oxide having a nitrogen content of 0.5 to 4 atom %.
9. The use of the composite body according to claims 1 to 8 as an element to be subjected to wear and for machining metallic and non-metallic materials. 35